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Resistance of blended concrete containing an industrial petrochemical residue to chloride ion penetration and carbonation

Resistencia a la penetración del ión cloruro y a la carbonatación de concretos adicionados, con un residuo de la industria petroquímica

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ABSTRACT

In this study, the resistance of blended concrete containing catalytic cracking residue (FCC) to chloride ion penetration and carbonation was examined. FCC was added at the levels of 10%, 20%, and 30% as partial replacement for cement. Concretes with 10% of silica fume (SF), 10% of metakaolin (MK), and without additives were evaluated as reference materials. The rapid chloride permeability test (RCPT) performed according to ASTM C1202 standards and an accelerated carbonation test in a climatic chamber under controlled conditions (23 °C, 60% RH and 4.0% CO_2), were used in order to evaluate the performance of these concretes. Additionally, their compressive strength was determined. The results indicate that binary blends with 10% FCC had similar compressive strength to concrete without additives and had lower chloride permeability. 10% SF and 10% MK exhibited better mechanical behavior and a significant decrease in chloride penetration when compared to 10% FCC. It is noted that there was an increase in concrete carbonation when FCC or MK were used as additives. It was also observed that with longer curing time, the samples with and without additives, presented higher resistance to carbonation. The accelerated corrosion test by impressed voltage was also performed to verify the findings and to investigate the characteristics of corrosion using a 3.5% NaCl solution as electrolyte. The mixtures that contained 10% FCC were highly resistant to chloride ion penetration and did not present cracking within the testing period.

Keywords: fluid catalytic cracking, metakaolin, silica fume, pozzolanic additions, blended concrete, carbonation resistance, chloride ion penetration.

RESUMEN

Se estudió la resistencia a la penetración del ión cloruro y a la carbonatación de concretos adicionados, con un residuo de catalizador de craqueo catalítico (FCC). El FCC fue adicionado en proporciones de 10%, 20% y 30%; como reemplazo parcial del cemento. Se evaluaron concretos como referencia con 10% de humo de sílice (HS), 10% de metacaolín (MK) y sin adición, así mismo, para evaluar el desempeño de estos concretos, se realizaron los ensayos de permeabilidad rápida a cloruros (PRC) de acuerdo con la norma ASTM C1202, y un ensayo de carbonatación acelerado bajo condiciones controladas (23 °C, 60% HR and 4.0% CO2). Adicionalmente, se determinó la resistencia a la compresión y de esta manera los resultados muestran que las mezclas binarias con 10% de FCC tuvieron una resistencia a la compresión, similar al concreto sin adición y más baja permeabilidad a los cloruros.

Las mezclas con 10% HS y 10% MK, mostraron un mejor comportamiento mecánico y significativo, se evidenció menor penetración de cloruros comparado con FCC al 10%. Se observó también, un incremento en la carbonatación del concreto cuando se utilizó adición de FCC o MK y es claro que a mayor tiempo de curado, las muestras con y sin adición, presentan mayor resistencia a la carbonatación. El ensayo de corrosión acelerado por voltaje impreso, fue realizado para investigar y verificar las características corrosivas, usando como electrolito una solución de NaCl al 3.5%. Las muestras con FCC al 10%, tuvieron una mayor resistencia a la penetración del ión cloruro, y el tiempo del ensayo no fue suficiente para iniciar un agrietamiento.

Palabras clave: catalizador de craqueo catalítico, metacaolín, humo de sílice, adiciones puzolánicas, concreto adicionado, resistencia a carbonatación y penetración de ión cloruro.

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Introduction

The factors primarily contributing to reinforcing steel corrosion are carbonation and chloride attack; therefore, these are responsible for a large share of the costs involved in rehabilitation of concrete structures.

Concrete carbonation is a process by which atmospheric carbon dioxide reacts with the calcium in calcium hydroxide and calcium silicate hydrate to form calcium carbonate. Once carbonation has taken place, the high pH of the concrete pore solution will begin to drop to as low as 9 or 8 for fully carbonated concrete (Pacheco-Torgal et al., 2012). As a result, the passive layer surrounding the steel reinforcement begins to deteriorate (Parrot, 1987). One of the principal factors that directly impact the process of carbonation is the water-to-cement ratio (w/c); it is well known that an increase of the w/c increases the depth of carbonation (Tsuyoshi et al., 2003). The relative humidity (RH) and the concentration of CO_2 are the most important environmental factors (Aguirre and Mejía de Gutiérrez, 2013). It has been reported that RH values between 50% and 75% promote the diffusion of CO_2 (Roberts, 1981) and that CO_2 concentration in the atmosphere may fluctuate from 0.03% in rural environments to over 0.3% in cities, where the incidence of carbonation is higher (Gruyaert et al., 2013; Pacheco-Torgal, 2012). For this reason, in tropical non-marine environments carbonation may be the main deterioration mechanism in reinforced concrete. Several researchers have attempted to correlate the mechanism of carbonation with concrete properties, particularly mechanical strength and porosity, having detected inconsistencies in the results, especially when blended concretes are used (Pacheco-Torgal, 2012). It should be noted that some authors agree that concrete containing supplementary cementitious materials are more susceptible to carbonation than ordinary Portland cement concrete (Ho and Lewis, 1987; Sideris et al., 2006; Papadakis, 2000; Gruyaert et al., 2013).

Chloride ions, from deicing salts or seawater, are the primary cause of reinforcing steel corrosion in highways and marine or coastal structures. Chlorides are transported through the concrete pore network, where they may be present as free ions or bound to cement hydration products in the form of Friedel´s salt or physically adsorbed to calcium silicate hydrates (Loser et al., 2010). Consequently, the chloride resistance mainly depends on the type of binder, the water-to-binder ratio, the hydration degree of the cement and of the supplementary materials present in the mixture.

Mejía de Gutiérrez et al. (2009) studied blended concretes with different types of MK and SF and made behavioral comparisons regarding carbonation and chloride ion penetration, finding a higher depth of carbonation in blended concretes when compared to control concrete. This tendency decreased as the curing age increased. On the other hand, these same materials exhibit better performance in the presence of chlorides.

A residue of the petrochemical industry called spent fluid catalytic cracking catalyst (FCC), has been studied in the last few years. This material presents pozzolanic characteristics comparable to those of metakaolin (Payá et al., 2001; Borrachero et al., 2002; Soriano et al., 2008; Trochez et al., 2010; Torres et al., 2012, 2012a, 2013). Zornoza et al. (2009) measured the chloride ingress (penetration) rate in mortars with FCC by means of thermogravimetric analysis and found that the pozzolanic reaction of FCC increases the hydrated calcium aluminates and silicoaluminates content, so the chloride binding capacity of mortars was highly improved, consequently the resistance to chloride-ion penetration increases.

This article presents the evaluation of the resistance against chloride penetration and carbonation of concretes with a partial substitution of cement by catalyst cracking residue (FCC) in proportions of 0, 10, 20 and 30%. The results are compared with blended concretes containing 20% Metakaolin (MK) and 10% Silica Fume (SF).

Materials and Experimental Procedure

An ordinary Portland cement (OPC) Type I was used for concrete preparation. Spent fluid catalytic cracking catalyst (FCC), Metakaolin (MK) and Silica Fume (SF) were used as supplementary materials. FCC was supplied by a Colombian petroleum company (Ecopetrol, Cartagena). The chemical and physical characteristics of these raw materials are shown in Table 1. The Average Particle Size was determined by laser granulometry in a Mastersizer 2000 particle size analyzer (Malvern Instrument). The pozzolanic activity was determined according to ASTM C618 standards. As shown in Table 1, FCC is composed almost entirely of silica and alumina, with a composition similar to that of Metakaolin. ASTM C618 standard requires a minimum pozzolanic activity index of 75% at 28 days of curing to consider a material as a pozzolan; this requirement is met as is shown in Table 1. All the supplementary materials used comply with that parameter.

Table 1.Chemical and physical characteristics of the FCC, MK, SF, and cement used.

Characterístics	Cement (OPC)	MK	SF	FCC
	Chemical Composition%			
SiO_2	19.43	53.38	96.90	43.97
Al_2O_3	4.00	43.18	0.21	45.48
Fe_2O_3	3.61	1.29	0.74	--
CaO	64.46	0.05	0.29	0.43
MgO	1.52	0.35	0.55	--
K_2O	0.39	1.11	1.16	0.15
TiO_2	0.34	0.59	0.59	0.69
Loss on Ignition	2.58	0.52	----	2.19
	Physical Properties			
Density (kg/m³)	3.13	2.50	2.60	2.63
Average Particle Size (μm)	16.07	7.53	0.18	18.00
Pozzolanic Activity index, 28 days	-	92.9	110	97.4

X-ray diffractometry was carried out using RX Rigaku RINT 2200 with copper lamp. Figure 1 shows the diffractograms (XRD) for FCC, MK and SF. The catalyst residue contains different crystalline phases, such as zeolite type faujasite (F) $Na_2[Al_2Si_{10}O_{24}].nH_2O$ with peaks located in 2θ = 6.19°, 15.6°, 23.58°), kaolinite (K) and quartz (Q). FCC catalyst also has a high content of amorphous (glassy) aluminosilicate phases because of the partial destruction of the zeolite structures in service. In the case of MK, a material of amorphous characteristics is shown by the uprising of the baseline in the region 2θ = 15 to 30° and the disappearance of the peaks corresponding to kaolinite. The intense broad peak observed for SF indicated that this material is totally amorphous.

Concrete mix design and tests

The concrete mix design resulted in a total binder content of 380 kg/m³ and a total amount of aggregate of 1727 kg/m³ (55% coarse and 45% fine), obtained by mixing coarse and fine aggregate respectively. The aggregates used are from alluvial origin. Coarse aggregate with a maximum nominal size of 12.7 mm, nominal density of 2668 kg/m³, unit weight of 1542 kg/m³ and absorption of 3.0%, and sand with nominal density of 2679 kg/m³, unit weight of 1667 kg/m³, absorption of 2.1% and a fineness modulus of 2.84 were used. Six concrete mixes including the control mix (without addition) were produced. Two mixtures were used as reference containing 20% MK and 10% SF. The other mixtures were made with partial substitution of OPC by FCC (10, 20 and 30%). A constant effective water-to-binder ratio (w/b) of 0.5 was selected (aggregates moisture corrections were included in calculations); this value is based on the durability requirements specified in the Colombian Earthquake Resistant Construction Code NSR-10, item C.4.2. In order to maintain the workability, a superplasticizer was used.

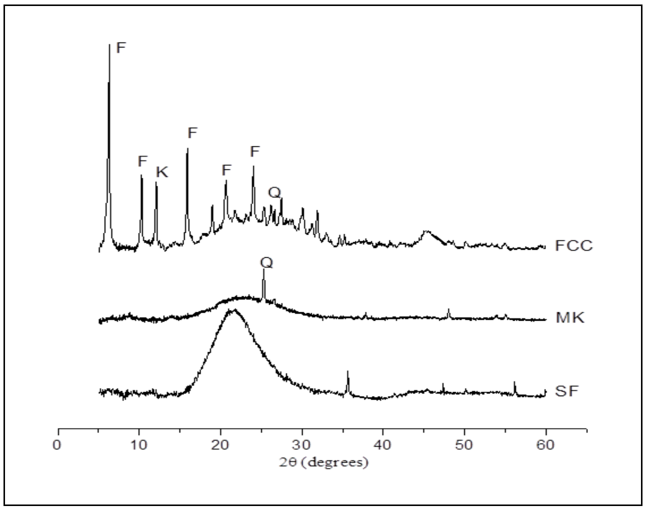


Figure 1. X-ray diffractograms of FCC, MK, SF (F: faujasite, Q: quartz, K: kaolinite).

The samples were cured in water saturated with $Ca(OH)_2$ at room temperature up to 360 days. Compressive strength properties and durability such as water absorption, chloride permeability and resistance to concrete carbonation were evaluated at different ages.

The water absorption and capillary absorptivity tests were carried out according to ASTM C642 (Standard Test Method for Density, Absorption and Voids in Hardened Concrete) and ASTM C1585 (Standard Test Method for Measurement of Rate of Absorption of Water for Hydraulic-Cement Concretes). The first test provides a measure of the total water permeable pore space and the second represents effective porosity or porosity that is accessible to water and therefore, to aggressive environmental agents.

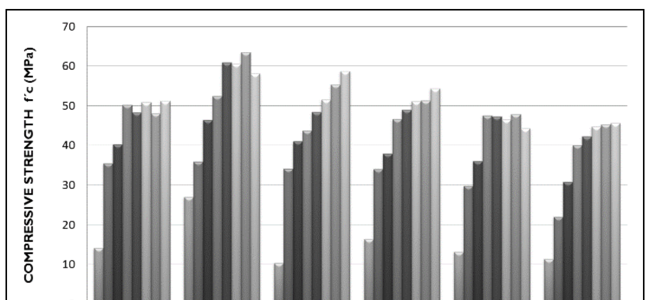
The chloride ion penetration was evaluated by means of the rapid chloride permeability test (RCPT), performed according to ASTM C1202 (Standard Test Method for Electrical Indication of Concrete's Ability to Resist Chloride Ion Penetration). The test consists of measuring the electrical current that passes through a concrete sample of a 100 mm diameter and 50 mm thickness maintaining the system at 60V for a period of 6 hours. Additionally, an accelerated corrosion test by impressed voltage was also performed to verify the findings and to investigate the characteristics of corrosion in terms of the initial current and the time of crack initiation. The specimens used for accelerated corrosion test were cylinders of 50 mm in diameter and 100 mm height with steel bars of 10 mm diameter placed in the middle of the specimens. Specimens were water cured and were then immersed in the corrosion cells. The electrolyte was 3.5% by weight of NaCl solution. A constant voltage of 5 V was applied between the anode (steel reinforcement) and the cathode and the current intensity was recorded up to the appearance of a longitudinal crack or a maximum period of 60 h. This test was realized in mortar samples (OPC and blended with FCC and MK) and the reinforcement was protected near the mortar surface in order to avoid corrosion at this point.

The study of carbonation was performed by an accelerated test using a climatic chamber under controlled conditions (23°C, 60% H.R. y 4.0% of CO_2) and cylindrical specimens of 75 mm in diameter and 150 mm height. The flat ends of these specimens were water proofed leaving the cylinder lateral faces uncovered and therefore, exposed to CO_2. The evaluation process was done by cutting off a 20 mm thick slice from specimens exposed to CO_2 and applying an indicator, phenolphthalein solution, over the flat surface. Since the color of the indicator turns purple at a pH above 9.0, non-carbonated zones are colored while carbonated zones are colorless. Six radial measurements of the zones that did not present coloration were taken for each specimen. The flat face of the same specimen was sealed again, with epoxy and the specimen was introduced into the chamber to measure the progression of the carbonation depth at later ages (3, 6 and 9 weeks) for each one of the curing ages.

Results and discussion

Compressive strength

Figure 2 presents the evolution of compressive strength for each of the mixtures after being cured for 1, 3, 7, 28, 56, 90, 120, 180 days. The testing was conducted according to the ASTM C39 standard using three cylinders with 100 mm in diameter and 200 mm of hieght, as samples.



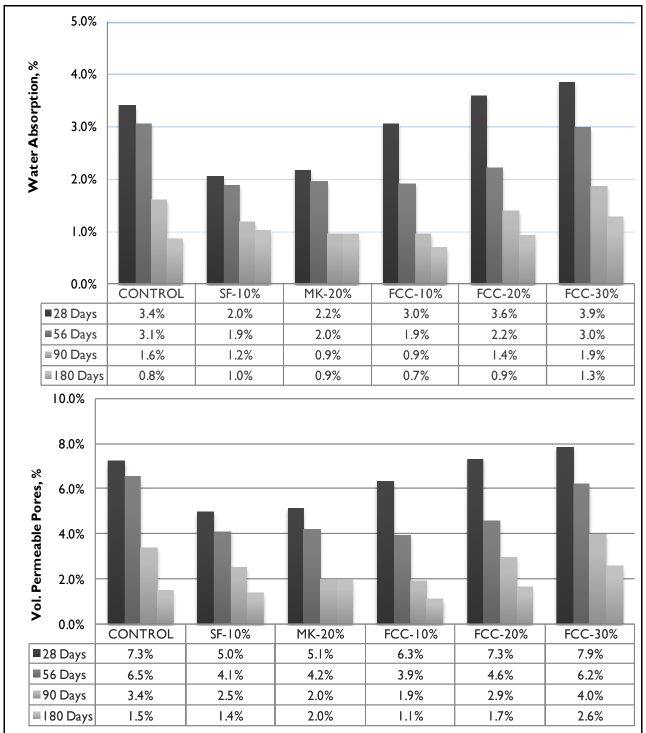
	CONTROL	SF-10%	MK-20%	FCC-10%	FCC-20%	FCC-30%
1 day	14.1	27.0	10.3	16.3	13.2	11.3
3 days	35.5	35.9	34.2	34.0	29.7	22.0
7 days	40.3	46.4	41.0	37.9	36.0	30.8
28 days	50.2	52.4	43.7	46.6	47.5	40.0
56 days	48.4	60.9	48.4	48.9	47.3	42.3
90 days	50.9	60.6	51.5	51.0	46.5	44.8
120 days	48.0	63.5	55.3	51.3	47.9	45.3
180 days	51.2	58.1	58.7	54.3	44.3	45.7

Figure 2. Evolution of compressive strength.

As expected, regardless of the type or percentage of addition, strength increases with age. It can be seen that at 1 day, the strength of the mixtures with FCC is higher than that of the mixture with MK and the strength of 10% FCC mixture is slightly higher than that of the control mixture. This may be due to the high reactivity of FCC reported at early ages, which is in line with other researchers' findings (Soriano et al., 2008; Payá et al., 2001, 2003; Antiohos et al., 2006). Figure 2 also shows that the resistance decreases with FCC additions higher than 20%, these results are consistent with those reported by Antiohos et al. (2006), highlighting that, the value at 28 days for all the mixes is higher than 40 MPa, allowing us to consider the concrete as structural concrete. In general, the optimal percentage of FCC is 10%. The maximum compressive strength for SF mixture (10%) was about 13.4% higher than that of the control mixture at 180 days and comparable to that of the concrete containing 10% MK at the same curing ages.

Water absorption, capillary absorptivity and Chloride ion permeability

ASTM C642 test results are presented in Figure 3. It can be appreciated that the concrete samples tested exhibit a water absorption of less than 5% and porosity of less than 10%. The absorptivity coefficient of blended concretes ranges between 0.10×10^{-2} and 0.15×10^{-2} kg/m^2 s$^{1/2}$. It can be seen that a longer curing period contributes to the improvement of cement hydration and pozzolanic reactions as well as create a denser microstructure with a smaller volume of capillary pores, resulting in a concrete with lower permeability.

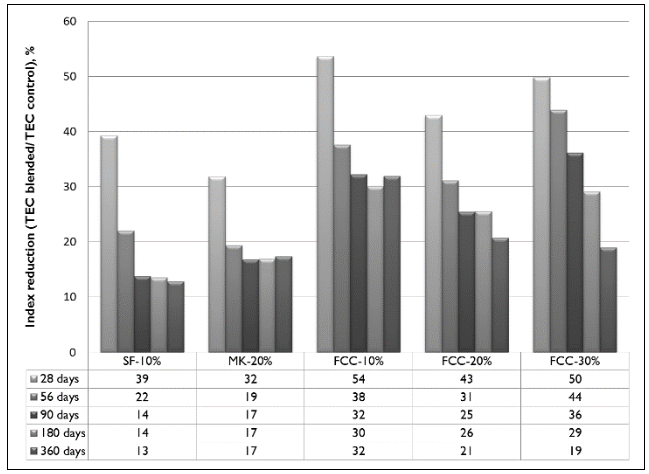


	CONTROL	SF-10%	MK-20%	FCC-10%	FCC-20%	FCC-30%
28 Days	3.4%	2.0%	2.2%	3.0%	3.6%	3.9%
56 Days	3.1%	1.9%	2.0%	1.9%	2.2%	3.0%
90 Days	1.6%	1.2%	0.9%	0.9%	1.4%	1.9%
180 Days	0.8%	1.0%	0.9%	0.7%	0.9%	1.3%

	CONTROL	SF-10%	MK-20%	FCC-10%	FCC-20%	FCC-30%
28 Days	7.3%	5.0%	5.1%	6.3%	7.3%	7.9%
56 Days	6.5%	4.1%	4.2%	3.9%	4.6%	6.2%
90 Days	3.4%	2.5%	2.0%	1.9%	2.9%	4.0%
180 Days	1.5%	1.4%	2.0%	1.1%	1.7%	2.6%

Figure 3. Water absorption according ASTM C642.

The chloride ion penetration was evaluated at different curing ages (28, 56, 90 and 180 days). The total electric charge (TEC, coulombs), passed through the samples, is calculated using the results obtained in the RCPT test. Figure 4 shows an index of reduction of the TEC, calculated as the percentage ratio between the TEC of blended material (OPC + addition %) and the TEC of the control sample for different curing ages. This figure shows that blended concretes present a higher resistance to chloride penetration than the control mixes and that this resistance increases with curing time. This could be attributed to the refinement of the matrix pore network resulting from the pozzolanic addition reaction. In general, the addition of FCC can reduce the penetration of chloride up to 54% at 28 days curing.



	SF-10%	MK-20%	FCC-10%	FCC-20%	FCC-30%
28 days	39	32	54	43	50
56 days	22	19	38	31	44
90 days	14	17	32	25	36
180 days	14	17	30	26	29
360 days	13	17	32	21	19

Figure 4. Chloride Permeability Test Results.

In natural exposure, especially chloride environments, the presence of high alumina pozzolan contributes additionally to the formation of Friedel's salt. This compound acts as a barrier to the chloride ingress through the cementitious matrix (Mejía de Gutierrez et al., 2000; Torres et al., 2007; Morozov et al., 2013). Zornosa et al. (2009) and Mejía de Gutierrez et al. (2000), using XRD and DTG (Differential Thermogravimetric analysis) techniques, confirm the formation of Friedel´s salt in mortars containing FCC and MK. It should be noted that the RCPT test takes only 6h, which is a very short period for allowing significant chloride binding. Therefore, this test is highly related to concrete electrical conductivity, which may be the parameter mainly affected by the addititives used.

Figure 5 presents the current intensity–time relationships for the reinforced mortar samples (control and blended with FCC and MK) evaluated by the impressed voltage test. These results are the average of two samples per mix. In general, current intensity with time. This increase is directly related to the progress of steel corrosion. Corrosion products generate tensile stresses in the hardened matrix leading to material cracking and to a significant increase of current intensity. It should be noted that the current passed through samples in this test is also an indication of the volume of corrosion products. The current intensity of the control mortar was superior to the other blended mixes. In general, the current intensity of blended mortars was very low indicating that these mixes were highly resistant to chloride penetration. Additionally, it can be observed that the use of FCC at 20% has a significant effect on the time of first crack appearance meanings a good corrosion resistance. The crack size was 1.053 mm for control mortar (100% OPC) and 0.496 for blended mortar with 10% FCC (figure 5). These results confirm those obtained in the RCPT test.

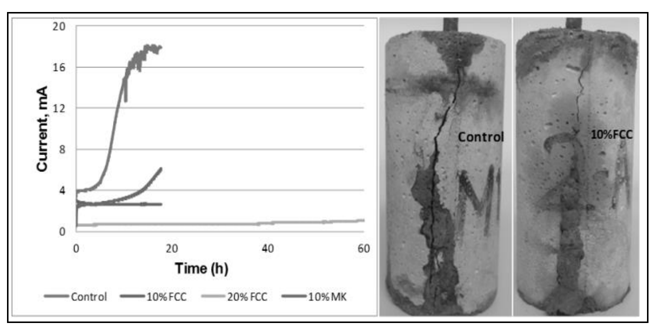


Figure 5. Accelerated Corrosion test.

Results also indicate that concretes with FCC are durable therefore, they can be used in reinforced concrete structures exposed to chloride-contaminated environments. According to these results, the recommended proportion is 20% FCC.

Resistance to concrete carbonation

The carbonation tests were performed at different curing ages (28, 56, 90 and 180 days). Figure 6 shows photographs of specimens cured for 28 days and exposed for 9 weeks to CO_2 inside the chamber after the application of phenolphthalein.

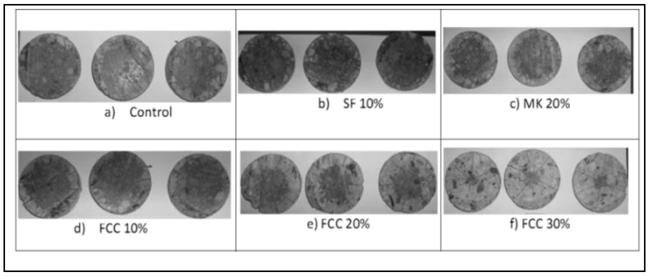


Figure 6. Carbonation depths.

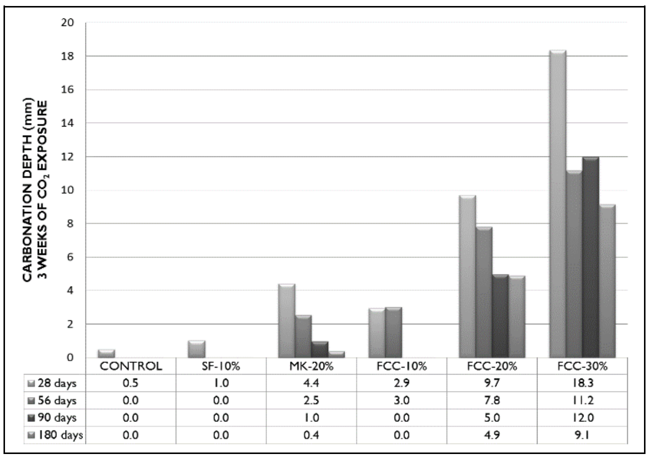


Figure 7. Carbonation depth at 3 weeks of exposure.

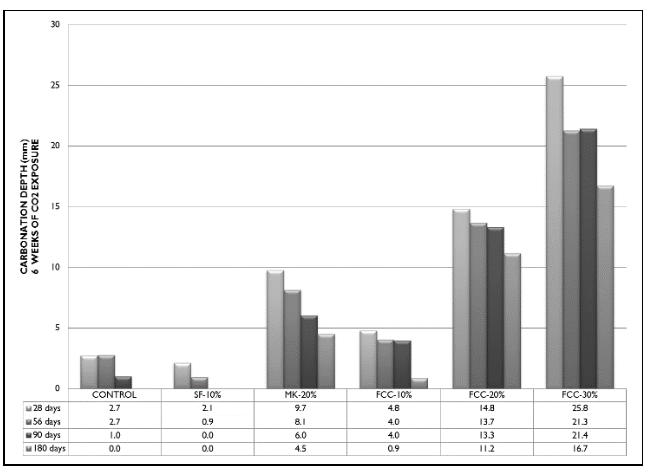


Figure 8. Carbonation depth at 6 weeks of exposure.

Figures 7 to 9 show that as the curing time increases, the carbonation depth decreases for all of the samples. The best behavior was presented by the control specimen and the blended samples with 10% SF. In regards to FCC concrete, the best behavior was presented by the sample with 10 % FCC, exhibiting better behavior than that of the specimen blended with MK. It can be seen that

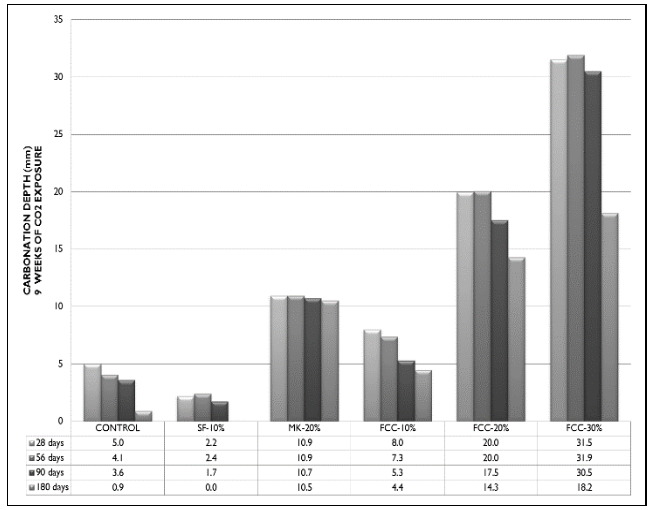


Figure 9. Carbonation depth at 9 weeks of exposure.

as the percentage of FCC increases, the susceptibility to carbonation increases. These results confirm that concrete containing supplementary cementitious materials such as FCC, MK or SF are more susceptible to carbonation than OPC concrete. It should also be noted that there is a wide range of published literature related to the carbonation of blended concrete and in some cases, contradictory findings are reported (Pacheco-Torgal et al., 2012). Some researchers report that the accelerated tests may underestimate the carbonation depths of specimens moist cured for 28 days, for this reason some standards recommend using specimens cured for 55 days (Gruyaert et al., 2013).

From these data, the carbonation coefficient (K_C) can be calculated using equation [1], where X represents the penetration depth (mm) and t, the exposure time. C is the CO_2 concentration used, 4%.

$$x = K_C\sqrt{t} \qquad (1)$$

Table 2. Estimated Carbonation depths (in mm) after 25 and 50 years exposure to a 0.03% or 0.3% CO_2 environment.

	Conditions		Carbonation accelerated test (mm/√year)					
	CO_2 %	Years	Control	SF-10%	MK-20%	FCC-10%	FCC-20%	FCC-30%
K_C	4%		11.0	5.6	27.1	17.8	46.9	75.9
Estimate of the Carbonation Depth (in mm) at different conditions								
x	0.03	50	6.7	3.4	16.6	10.9	28.7	46.5
x	0.03	25	4.8	2.4	11.8	7.7	20.3	32.9
x	0.30	50	21.2	10.8	52.6	34.5	90.8	146.9
x	0.30	25	15.0	7.7	37.2	24.4	64.2	103.9

The accelerated test method could provide an indication of the long-term concrete carbonation resistance. Considering that in actual exposure conditions, the level of CO_2 in the atmosphere varies from 0.03% to 0.3%, the carbonation depth of a concrete element after a service life of 25 and 50 years can be estimated using the equation [2] (Castro et al., 2004). In this equation, K_C and K_N represent the carbonation coefficient corresponding to two different concentrations of CO_2 named C and N. Using data from the accelerated test for specimens cured for 28 days, estimations of carbonation depths at 25 and 50 years were made (Table 2).

$$K_C/K_N = \sqrt{C/N} \quad (2)$$

Results show that in general, for environments containing 0.03% CO_2, the carbonation depth of blended concretes with SF, MK and FCC 10%, after 25 and 50 years, is lower than 20 mm. Taking into account the results presented in Table 2, concrete mixes with 20 and 30% of FCC would not perform well in a 0.3% CO_2 environment.

Conclusions

Given the results of this study, the following conclusions can be made: the catalyst residue obtained from the Colombian petroleum company plant is a pozzolan that substantially improves the mechanical properties and durability of concrete. It is a pozzolan of high reactivity at early ages, which leads to low chloride permeability. The behavior of FCC is comparable to that of metakaolin, a pozzolanic material well known worldwide. In general, blended concretes have greater susceptibility to carbonation, so it is recommended that they undergo an initial curing process. The optimal percentage of FCC is 10%, but in chloride environments, it is recommendable to use 20%.

The results obtained in this study allow us to conclude that the FCC is an additive that could compete in the market with other types of pozzolans, contributing positively to the strength and durability of concrete as well as environmental sustainability..

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